भारतीय मानक **IS 12846 : 2023**

Indian Standard

बोरिक एसिड, फोटोग्राफिक ग्रेड — विशिष्टि

(पहला पुनरीक्षण)

Boric Acid, Photographic Grade — Specification

(First Revision)

ICS 37.040.30

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

June 2023 **Price Group 7**

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

There are two Indian Standards available for boric acid. IS 263 : 1990 ‘Specification for boric acid for cosmetic industry (*fifth revision*)’ covers the requirements for boric acid for cosmetic industry and IS 10116 : 2015 ‘Specification for boric acid (*first revision*)’ covers the requirements for boric acid for use in manufacture of capacitors and in electronics, nuclear energy, foundries, fire proofing of workman’s apparel glass and ceramics, explosives, refractories, paints, chemicals, dye intermediates, electroplating, leather, adhesives and enamel industries. Since the requirements for photographic industry are different from those covered in the above standards, this standard was formulated to cover the requirements of boric acid for photographic industry.

Boric acid is used in the processing of sensitized photographic materials. It has a chemical formula of H_3BO_3 and molecular mass as 61.83.

This Standard was first published in 1989. In this revision following changes have been done:

a) Incorporation of alternate instrumental test method (AAS) for lead (Pb), iron (Fe) and arsenic (As); and
b) Incorporation of alternate spectrophotometric method for Pb and Fe.

The composition of the committee responsible for formulation of this standard is listed in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BORIC ACID, PHOTOGRAPHIC GRADE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for boric acid, photographic grade used in the processing of sensitized photographic materials.

2 REFERENCES

The standards listed in Annex A contains provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 REQUIREMENTS

3.1 Physical Appearance

The material shall be in the form of white free flowing crystals, free from lumps and external visible impurities.

3.2 Appearance of Aqueous Solution

5 g of the material dissolved in 100 ml of water shall be clear and colorless, and free from extraneous impurities.

3.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to methods given in Annex B. Reference to the relevant clauses of Annex B is given in col (4) of Table 1.

Table 1 Requirements for Boric Acid, Photographic Grade
(*Clause* 3.3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Assay (as H_3BO_3) percent by mass, *Min*	99.5	**B-2**
ii)	Iron (as Fe), percent by mass, *Max*	0.001	**B-3**
iii)	Heavy metals (as Pb), percent by mass, *Max*	0.001	**B-4**
iv)	Ammoniacal silver nitrate test	To pass the test	**B-5**
v)	Arsenic, percent by mass, *Max*	0.003	**B-6**

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable air-tight containers as agreed to between the purchaser and the supplier.

The containers shall be marked with the following information:

a) Name and grade (photographic) of the material;
b) Net mass of the material;
c) Month and year of manufacture;
d) Indication of the source of manufacture; and
e) Batch number or lot number in code or otherwise to enable the batch of manufacture to be traced from the records.

4.2 *BIS Certification Marking*

The containers may also be marked with the BIS Certification Mark.

4.2.1 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Annex C.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
IS 265 : 2021	Hydrochloric acid specification (*fifth revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 2088 : 1983	Methods for determination of arsenic (*second revision*)
IS 4905 : 2015	Random sampling and randomization procedures (*first revision*)
IS 11124 : 1984	Method for atomic absorption spectrophotometric determination of arsenic
IS 12074 : 1987	Method for determination of lead by atomic absorption spectrophotometry
IS 13320 : 1992	Determination of iron by atomic absorption spectrometry — Test method

ANNEX B

(*Clause* 3.3)

METHODS OF TEST FOR BORIC ACID, PHOTOGRAPHIC GRADE

B-l QUALITY OF REAGENTS

B-l.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

B-2 ASSAY (as H_3BO_3)

B-2.1 Reagents

B-2.1.1 Glycerine

B-2.1.2 Sodium Hydroxide, 1 N.

B-2.1.3 Phenolphthalein.

B-2.2 Procedure

Weigh accurately about 2.5 g of sample into a conical flask. Dissolve in 50 ml of water. Then add 50 ml of glycerine neutralized to phenolphthalein indicator, titrate with 1 N sodium hydroxide (NaOH) solution using phenolphthalein as indicator.

B-2.3 Calculation

Assay (as H_3BO_3)

$$\text{percent by mass} = \frac{V \times N \times 6.183}{M} \quad \ldots.(1)$$

where

V = volume of NaOH solution consumed;
N = normality of NaOH solution; and
M = mass of the sample taken for analysis.

B-3 DETERMINATION OF IRON

B-3.1 General

Three methods for the determination of Iron are prescribed. Either of these may be used for general routine purposes, but in case of a dispute Method B shall be used as referee method.

B-3.2 Method A

B-3.2.1 *Principle*

The dissolved iron under slightly acidic medium is reduced using hydroxyl ammonium chloride and the color is developed with 2,2'-bipyridyl solution [iron (II)-2,2' bipyridyl complex]. The absorbance of the color is measured by a spectrophotometer at 522 nm.

B-3.2.2 *Apparatus*

B-3.2.2.1 Spectrophotometer suitable for measurement at 522 nm, with accuracy of 1 nm.

B-3.2.2.2 Standard laboratory glass apparatus

B-3.2.3 *Reagents*

B-3.2.3.1 *Distilled water*

B-3.2.3.2 *Hydrochloric acid*, approximately 6 mol/l (1:1 *v/v*).

B-3.2.3.3 *Hydrochloric acid*, approximately 1 mol/ l.

To 910 ml of distilled water, add 90 ml of concentrated hydrochloric acid and mix.

B-3.2.3.4 *Hydroxylammonium Chloride*

Dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCI$) in water and dilute to 100 ml.

B-3.2.3.5 *Ammonium acetate solution, 30* percent (m/v).

B-3.2.3.6 *2,2'-Bipyridyl solution*

Dissolve 1 g of 2,2'-Bipyridyl in 10 ml of hydrochloric acid solution (1 mol/l) and dilute to 100 ml.

B-3.2.3.7 *Standard iron solution A*

Dissolve 0.702 2 g of ferrous ammonium sulphate hexahydrate [$FeSO_4NH_4SO_4.6H_2O$] in water in a 250 ml beaker, add 50 ml of sulphuric acid (about 1 mol/l) and mix. Transfer quantitatively into a 1 li volumetric flask, make up to the mark and mix well. (1 ml = 0.1 mg of Fe)

B.2.3.8 *Standard iron solution B*

Dilute 100 ml of the above solution (solution A) and dilute to 1 l in a volumetric flask, mix well. (1 ml = 10 µg of Fe)

B-3.2.4 *Procedure*

B-3.2.4.1 *Calibration*

Into each of a series of level 100 ml volumetric flasks, place the quantities of standard iron solution B as given Table 2:

Sl No. (1)	*Standard Iron Solution. B (ml)* (2)	*Corresponding Iron Content as Fe µg* (3)
i)	0 (Compensation)	0
ii)	5.0	50
iii)	10.0	100
iv)	15.0	150
v)	20.0	200
vi)	25.0	250
vii)	30.0	300
viii)	35.0	350
ix)	40.0	400
x)	45.0	450
xi)	50.0	500

Add to each volumetric flask an amount of water sufficient to dilute to 50 ml, then 2 ml of hydrochloric acid and 2 ml of hydroxylammonium chloride solution and after 5 min, add 5 ml of ammonium acetate solution and 1 ml of 2,2' bipyridyl solution. Dilute to mark, mix well, and wait for 10 min. Measure absorbance of each solution using spectrophotometer with l cm cell at 522 nm against reagent blank (containing equal amount of added reagent) and make up the volume to 100 ml.

B-3.2.4.2 *Preparation of calibration graph*

Prepare a calibration graph with iron as Fe in l.0 µg/100 ml as abscissa and absorbance as ordinates.

B-3.2.4.3 *Determination of iron*

Weigh accurately to the nearest 1 mg, an amount of Boric Acid sample containing 100 µg to 1 000 µg of iron (as Fe) into a platinum dish. Wet it in minimum amount of distilled water and neutralize with 6 mol/l hydrochloric acid solution and add a few drops in excess. Evaporate to dryness on a water bath. Wet it with a few drops of dilute hydrochloric acid and then dissolve in 20 ml to 30 ml of distilled water and quantitatively transfer into a l00 ml one-mark volumetric flask, dilute to mark and mix well. If the solution is turbid filter through a Whatman No. 541 filter paper and discard 10 ml to 15 ml of the initial filtrate.

Transfer a suitable aliquot volume (or the filtrate if filtered) containing 50 µg to 500 µg of Fe into a l00 ml one-mark volumetric flask. Dilute to around 50 ml (if less), add 2 ml hydrochloric acid and 2 ml of hydroxylammonium chloride. Mix well and after 5 min add 5 ml of ammonium acetate solution followed by 1 ml of 2,2'-bipyridyl solution. Dilute to mark, mix well and allow to stand for 10 min. Measure the absorbance using spectrophotometer at 522 nm wave length as described for calibration. Use the same cell size as the one used for calibration and use blank test solution as reference.

B-3.2.4.4 *Blank test*

Prepare a blank test solution using same procedure as used for determination of iron but excluding Boric acid sample.

B-3.2.5 *Calculation and Expression of Results*

By means of the calibration graph determine the concentration of iron in micrograms corresponding to the absorbance value of the test solution. Calculate the iron content as-

Iron (as Fe), percent by mass =

$$\frac{A \times 100 \times 100}{E} \qquad \ldots(2)$$

where

A = mass in g of iron determined in the sample solution, and

E = mass in g of the sample taken for the test.

B-3.3 Method B

B-3.3.1 Reagent*s*

B-3.3.1.1 *p*H *5 Acetate buffer*

Add 23 g of anhydrous sodium acetate to 58 ml of 2 molar acetic acid and dilute to 1 l with water. Adjust the final *p*H of the solution to 5.0 ± 0.l with acetic acid or 10 percent sodium hydroxide (NaOH) solution.

B-3.3.1.2 *o-phenanthroline mixture*

Thoroughly mix equal parts of 0.1 percent aqueous solution *o*-phenanthroline, 10 percent hydroxylamine hydrochloride solution in water and *p*H 5 acetate buffer.

B-3.3.1.3 *Standard iron solution*

Dissolve 0.70 g of ferrous ammonium sulphate $[Fe(NH_4)_2(SO_4)_2.6H_2O]$ in 500 ml of water and add 20 ml of dilute sulphuric acid (1 : 5). Dilute to 1 000 ml and mix thoroughly. Dilute 10 ml of this solution to 100 ml with water (1 ml = 0.000 01 g Fe).

B-3.3.2 *Procedure*

Dissolve 1 g sample in 40 ml of water. Add 5 ml of the *o*-phenanthroline mixture and let stand for 10 min. Dilute to 50 ml with water and mix well. Any color produced shall not be greater than that produced by a freshly prepared solution of a soluble iron salt equivalent to 0.01 mg of iron containing 2 ml of 1 percent (*v/v*) hydrochloric acid (HCl), and treated in the same manner as that of sample. Use Nessler cylinders for comparison. The sample shall be considered to have passed the requirement of the test if the colour produced with the sample is not darker than that with the standard solution.

B-3.4 Method C

Determine iron content by Atomic Absorption Spectrophotometer (AAS) in accordance with method given in IS 13320.

B-4 DETERMINATION OF HEAVY METALS

B-4.1 General

Three methods for the determination of Heavy Metals are prescribed. Either of these may be used for general routine purposes, but in case of a dispute Method B shall be used as referee method.

B-4.2 Method A

B-4.2.1 *Principle*

An acidified sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent (pH 10 to pH 11.5). The quantity of lead present in the sample is determined spectrophotometrically — by measuring the absorbance at 510 nm of the chloroform extract containing the lead dithizonate complex.

B-4.2.2 *Minimum Detection Limit*

1.0 µg Pb/10 ml dithizone solution (extract).

B-4.2.2 *Apparatus*

Spectrophotometer for use at 510 nm with a path length of 1 cm or longer;

*p*H meter;

Standard Volumetric Glasswares;

TEF Beaker, 100 ml for Acid Digestion;

Separatory Funnels, 250 ml and 500 ml;

All glasswares are to be cleaned with 1:1 HNO_3, and rinsed thoroughly with distilled water.

B-4.2.3 *Reagents*

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

B-4.2.3.1 *Stock lead solution*

Dissolve 0.159 9 g lead nitrate $[(Pb(NO_3)_2$, minimum purity, 99.5 percent (*w/w*)] in about 200 ml of water. Add 10 ml concentrated HNO_3 and dilute to 1 000 ml with water. (1.0 ml = 100 µg of Pb).

B-4.2.3.2 *Standard lead solution*

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

Nitric Acid — Concentrated (18N);

Nitric Acid — Dilute —20 percent, *v/v*;

Ammonium Hydroxide — Concentrated (14N);

Ammonium Hydroxide — Dilute 10 percent, *v/v* and 1 percent, *v/v*; and

Citrate-Cyanide Reducing Solution – Dissolve 200 g anhydrous ammonium citrate [$(NH_4)_2$ $HC_6H_5O_7$] 10 g anhydrous sodium sulphite (Na_2SO_3), 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with 1 l of concentrated NH_4OH.

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

B-4.2.3.3 *Stock dithizone solution*

Dissolve 25 mg dithizone in about 50 ml chloroform ($CHCl_3$) taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (*v/v*) NH_4OH solution, shake moderately for about 1 min. Transfer the $CHCl_3$ layer to another 250 ml (separating) separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the $CHCl_3$ layer) with 100 ml of 1 percent (*v/v*) NH_4OH solution, transfer the $CHCl_3$ layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1:1 HC1 in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of $CHCl_3$. Dilute the combined extract to 250 ml with $CHCl_3$, 1 ml of this solution will contain 100 µg of dithizone.

B-4.2.3.4 *Working dithizone solution*

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with $CHCl_3$, 1 ml of this solution will contain 40 µg of dithizone.

B-4.2.4 *Procedure*

Sample Digestion

Digest all samples for lead as per standard digestion procedure using HNO_3 H_2SO_4 and HNO_3-$HClO_4$.

To 100 ml acidified sample (*p*H = 2) add 20 ml of dilute (20 percent, *v/v*) HNO_3, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30 s, allow to stand (to get two separate layers). Discard 1 ml to 2 ml $CHCl_3$ layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

Calibration Curve

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

B-4.2.5 *Calculation*

mg Pb/l = µg (in 10 ml extract obtained from calibration curve)/Volume of sample (ml)

B-4.3 Method B

B-4.3.1 *Apparatus*

B-4.3.1.1 *Nessler cylinders, 50 ml capacity.*

B-4.3.2 *Reagents*

B-4.3.2.1 *Hydrochloric acid* (*see* IS 265).

B-4.3.2.2 *Ammonium hydroxide solution* (1 : 2)

B-4.3.2.3 *p-nitrophenol indicator*

B-4.3.2.4 *Standard lead solution*

Prepare a stock solution by dissolving 1.600 g of lead nitrate in water, add 1 ml of concentrated nitric acid and dilute to 1 000 ml with water. Further dilute 10 ml of this solution to 1 000 ml with water (1 ml = 0.000 01 g Pb).

NOTE — The dilute solution shall be freshly prepared since it deteriorates on standing.

B-4.3.3 *Procedure*

Dissolve 2 g of sample in 40 ml of water. Add 2 ml of 1 percent (*v/v*) hydrochloric acid (HCl) and 5 ml of hydrogen sulphide (H_2S) water, Dilute to 50 ml with water and mix well. Any colour produced shall not be darker than that produced in a solution containing 0.02 mg of lead, 2 ml of 1 percent (*v/v*) hydrochloric acid (HCl), 5 ml of hydrogen sulphide (H_2S) water and diluted to 50 ml with water. Use Nessler cylinders for comparison. The sample shall be considered to have passed the requirement of the test if the color produced with the sample is not darker than that with the standard solution.

B-4.4 Method C

Determine lead content by atomic absorption spectrophotometer in accordance with method given in IS 12074.

B-5 AMMONIACAL SILVER NITRATE TEST

B-5.1 Reagent

Mix equal volumes of 10 percent silver nitrate solution and concentrated ammonium hydroxide solution.

B-5.2 Procedure

Dissolve 1 g of sample in 20 ml. of water. Add 10 ml of freshly prepared reagent and mix well. Let stand for 2 min and compare with a blank prepared by adding 5 ml of concentrated ammonium hydroxide solution to a solution of 1 g of sample dissolved in 25 ml of water. No colour or precipitate in excess of that produced in the blank should be noticeable.

NOTE — Both the sample and the blank should be prepared simultaneously and discarded immediately after the test.

B-6 DETERMINATION OF ARSENIC

B-6.1 Two methods for the determination of Arsenic are prescribed. Either of these may be used for general routine purposes, but in case of a dispute Method A shall be used as referee method.

B-6.2 Method A

B-6.2.1 *Procedure*

Dissolve exactly 0.5 g of sample and 1 g of citric acid in 30 ml of water and proceed as per IS 2088. The material shall be considered to have passed the test if the arsenic content does not exceed 0.003 percent by mass.

B- 6.3 Method B

Determine arsenic content by atomic absorption spectrophotometer (AAS) in accordance with method given in IS 11124.

ANNEX C

(*Clause* 5.1)

SAMPLING OF BORIC ACID, PHOTOGRAPHIC GRADE

C-l LOT

In a consignment all the containers of the same grade of material and produced by one manufacturer shall constitute a lot. Each lot shall be separately inspected for deciding the conformity to the requirements of the specification.

C-2 SCALE OF SAMPLING

The number of containers to be selected from a lot shall be in accordance with Table 3 and these shall be selected at random. In order to ensure the randomness of selection, procedures given in IS 4905 may be followed.

C-3 For description as well as appearance of solution each container of the sample shall be opened and examined for relevant requirements. For other characteristics, a composite sample shall be prepared by taking adequate quantity from each of the sample containers and thoroughly mixing them by suitable means. This composite sample shall be tested for the relevant requirements given in Table 1.

C-4 The lot shall be considered conforming to the requirements of this specification if both individual as well as composite samples pass the corresponding tests.

Table 3 Scale of Sampling
(*Clause* C-2)

Sl No. (1)	**Lot size** (2)	**Sample size** (3)
i)	(*N*)	(*n*)
ii)	up to 25	2
iii)	26 to 50	3
iv)	51 to 100	4
v)	101 and above	5

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Electroplating Chemicals and Photographic Materials Sectional Committee**,** CHD 05

Organization	*Representative(s)*
CSIR - Central Electrochemical Research Institute, Karaikudi	Dr B. Subramanian, (***Chairperson***)
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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in

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Amendments Issued Since Publication

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002 — 2323 7617

Eastern : 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091 — 2367 0012, 2320 9474

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019 — 265 9930

Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113 — 2254 1442, 2254 1216

Western : Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093 — 2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.

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